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Short communication

Low-cost method for sodium borohydride regeneration and the energy efficiency of its hydrolysis and regeneration process

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HIGHLIGHTS

- The regeneration process for $NaBH_4$ is designed using MgH_2 with $NaBO_2$.
- The energy efficiency of the hydrolysis and regeneration of $NaBH_4$ is 49.91%.
- A cheap method for $NaBH_4$ regeneration was developed by reacting H–Mg_3La with $NaBO_2$.
- The mechanism of $NaBH_4$ regeneration by reacting Mg_3La hydride with $NaBO_2$ is revealed.

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ABSTRACT

Hydrolysis of sodium borohydride ($NaBH_4$) is one of the most attractive methods for energy generation of mobile systems used as hydrogen source because of the high gravimetric density and controllable hydrogen generation of $NaBH_4$. However, regeneration of $NaBH_4$ is a key issue that remains to be solved, and the energy efficiency of $NaBH_4$ is unknown. In the present study, the energy efficiency of $NaBH_4$ hydrolysis and the entire process of sodium metaborate ($NaBO_2$) regeneration via reaction with magnesium hydride (MgH_2) is determined through thermodynamics calculations. The maximum energy efficiency is 49.91%, indicating that $NaBH_4$ generation by reaction between MgH_2 and $NaBO_2$ during ball milling is feasible. An inexpensive high-energy ball milling method is employed to regenerate $NaBH_4$ by reaction of $NaBO_2$ with magnesium–lanthanum hydrides (H–Mg_3La). Products after ball milling are characterized through Fourier transform infrared spectroscopy and X-ray diffraction measurements. In the reaction of $NaBO_2$ with H–Mg_3La, MgH_2 reacts with $NaBO_2$ and then lanthanum hydride (LaH_3) reacts with $NaBO_2$ to produce $NaBH_4$.

1. Introduction

Because of the world energy crisis, replacement of fossil fuel has become a key issue. In this regard, hydrogen energy is an important alternative source of energy [1]. Unlike oil or natural gas, however, hydrogen is an energy carrier rather than a source of energy [2]. Appropriate methods for hydrogen generation and storage must therefore be developed to utilize it [3]. Hydrolysis is one of the most attractive methods of hydrogen generation because it obviates storage and produces a large amount of hydrogen. Among the hydrogen complexes that produce hydrogen by hydrolysis and function as storage material for hydrogen, sodium borohydride ($NaBH_4$) has been extensively studied. It has been utilized in hydrogen supply systems of fuel cells [4,5]. The nonhazardous characteristic and high gravimetric density (10.8wt%) [3] of $NaBH_4$ favor the use of this complex in hydrogen production. $NaBH_4$ hydrolyzes according to the following process:

$$NaBH_4 + 2H_2O \rightarrow NaBO_2 + 4H_2 \quad \Delta H = -75\ kJ\ mol^{-1}\ H_2 \qquad (1)$$

This highly controllable reaction generates pure hydrogen. Thus, it can be directly used in fuel cells [3,6]. The byproduct of this reaction, sodium metaborate ($NaBO_2$), is environmentally friendly

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and nontoxic. As $NaBH_4$ hydrolysis is irreversible, a key issue is discovering a means to convert $NaBO_2$ back to $NaBH_4$ [7]. For example, the less-expensive reducing metal, magnesium (or its hydride), has been used to produce $NaBH_4$ from dehydrated $NaBO_2$. Work on this approach was largely conducted by Kojima et al. [8]. They synthesized $NaBH_4$ by heating a mixture of dehydrated $NaBO_2$ and MgH_2 or a mixture of $NaBO_2$ and Mg under high H_2 pressure and elevated temperature. This synthesis proceeds through reactions described in Equations (2) and (3).

$$NaBO_2 + 2MgH_2 \rightarrow NaBH_4 + 2MgO \quad (2)$$

$$NaBO_2 + 2Mg + 2H_2 \rightarrow NaBH_4 + 2MgO \quad (3)$$

Other researchers further studied processes for converting $NaBO_2$ back to $NaBH_4$ through the above reaction [10,11]. Kojima et al. [8] synthesized $NaBH_4$ by heating dehydrated $NaBO_2$ and magnesium silicide (Mg_2Si) under high H_2 pressure at elevated temperature. However, energy consumption of such processes is very high. To achieve a new, economical route of $NaBH_4$ synthesis, Hsueh et al. [7], Çetin et al. [9], and Kong et al. [6] ball-milled dehydrated $NaBO_2$ and MgH_2 at room temperature. The yield of this process is 76%.

The U.S. Department of Energy advises against the use of $NaBH_4$ in on-board automotive hydrogen storage. One of the main reasons behind this advisory is the cost of $NaBH_4$ and the irreversible process of its hydrolysis [12]. We thus examined the energy efficiency and heat effect of the entire process of $NaBH_4$ recycling to determine the feasibility of hydrogen generation by $NaBH_4$ hydrolysis. $NaBO_2$ and MgH_2 were used to synthesize $NaBH_4$ by ball milling, and the energy efficiency of the entire recycling process was determined through thermodynamic calculations. The energy consumption of the regeneration procedure was discussed in accordance with the calculations. To reduce the cost and temperature of $NaBH_4$ synthesis, we reacted MgH_2 and lanthanum hydride (LaH_3) mixtures produced by hydrogenating magnesium–lanthanum alloy (Mg_3La) [13–15] with $NaBO_2$ by ball milling at room temperature. Our process avoids the use of MgH_2, which is synthesized by hydrogenation at a high temperature; it is thus an alternative route for the regeneration of $NaBH_4$ for industrial use.

2. Experimental

2.1. Sample preparation

MgH_2 powder (98% purity) was purchased from Alfa Aesar (USA). Mg_3La was prepared by induction melting of Mg (99.9%) and lanthanum (99.9%) in an alumina crucible under an argon atmosphere. The alloys were milled for 0.5 h in a QM-2SP planetary ball mill at a ball-to-powder mass ratio of 20:1. The $NaBO_2$ powder was dried at 280 °C to obtain anhydrous $NaBO_2$. To prevent samples and raw materials from oxidation and/or hydroxide formation, they were stored and handled in an Ar-filled glove box equipped with a recirculation system.

2.2. Synthesis of $NaBH_4$

Hydrogenation of Mg_3La was performed for 0.5 h at room temperature. MgH_2–$NaBO_2$ mixtures (2:1 mole ratio) and magnesium hydride–lanthanum hydride ($3MgH_2$–LaH_3)–$NaBO_2$ mixtures (4.4:9 mole ratio) were prepared. The mixtures were processed in a high-speed vibrating mill (QM-3C) using two sizes of balls.

2.3. Purification of $NaBH_4$

Purification of $NaBH_4$ was accomplished by extracting $NaBH_4$ with anhydrous ethylenediamine (99% purity) from the products after milling and then separating the extracted solution from the byproducts and remaining reactants through a polytetrafluoroethylene filter. The filtrate was dried in a vacuum oven at 50 °C to obtain $NaBH_4$.

2.4. Sample characterization

H–Mg_3La, as well as products after reaction and after purification were characterized by using a Philips X'Pert MPD X-ray diffractometer with Cu Kα radiation. Patterns in the 2θ range of 10°–90° were recorded at a scanning rate of 0.02° s^{-1}. The reaction products were analyzed by Fourier transformed infrared (FT-IR) spectroscopy (Bruker Vector33).

3. Results and discussion

3.1. Regeneration of $NaBH_4$ using $NaBO_2$ and MgH_2

To obtain a cyclical process with $NaBH_4$ hydrolysis and regeneration for hydrogen generation, $NaBH_4$ was regenerated by using $NaBO_2$ and MgH_2. Fig. 1 presents X-ray diffraction (XRD) patterns of the products after ball milling for different durations. Peaks of the XRD pattern of the product after 0.5 h of ball milling (Fig. 1(a)) could be indexed to MgH_2, $NaBH_4$ [16], and MgO. According to the phase analysis mentioned above, $NaBH_4$ and the by-product MgO were produced after 0.5 h of ball milling. Peaks of the XRD pattern of the product after 2 h of ball milling (Fig. 1(c)) could be indexed to $NaBH_4$ and MgO. In contrast to the XRD patterns in Fig. 1(a) and (b), the pattern in Fig. 1(c) does not have diffraction peaks of MgH_2. Stronger diffraction peaks of $NaBH_4$ in Fig. 1(c) compared with peaks in Fig. 1(d) suggest that part of the MgH_2 phase reacted with $NaBO_2$ and part of it became refined. Peaks of the XRD pattern of the product after 4 h of ball milling (Fig. 1(e)) could be indexed to $NaBH_4$ and MgO. The energy input for the vibrating mill used in this process of $NaBH_4$ regeneration was omitted in the subsequent calculation.

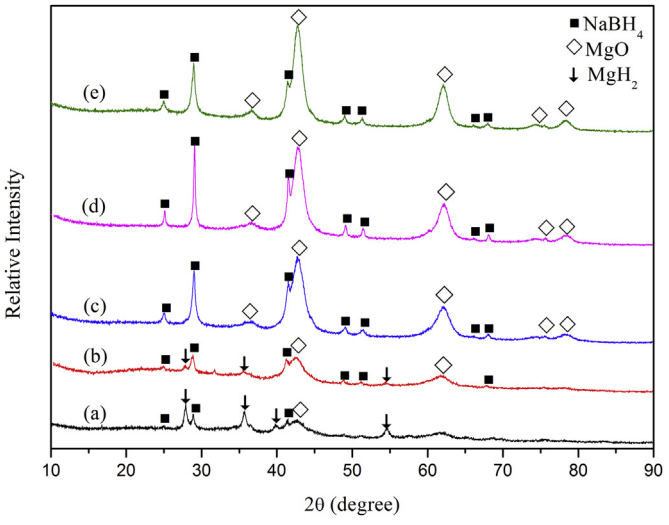

Fig. 1. XRD patterns of the powders produced after shaker milling the MgH_2–$NaBO_2$ mixture (in 2:1 mol ratio) for different durations (a) 30 min (b) 1 h (c) 2 h (d)3 h (e) 4 h.

3.2. Energy efficiency of $NaBH_4$ hydrolysis and regeneration

Calculation of the energy efficiency of $NaBH_4$ hydrolysis and regeneration was based on the synthesis of $NaBH_4$ from MgH_2 and anhydrous $NaBO_2$. The feasibility of this regeneration process was discussed according to the calculation results. Fig. 2 shows the cycle of $NaBH_4$ hydrolysis and regeneration of $NaBH_4$ and MgH_2. The energy efficiency is confirmed by the ratio of energy output (E_o) of the cycle to the energy (E_i) input for the reaction of the cycle.

To calculate the energy efficiency, the cycle was divided into three parts. First, $NaBH_4$ in contact with ruthenium catalyst hydrolyzes at room temperature [17], as described in Equation (1). Through a reaction described in Equation (4), the hydrogen generated from this process can be used in a fuel cell.

$$H_2 + 1/2O_2 \rightarrow H_2O \quad (4)$$

Second, the byproduct $NaBO_2$ reacts with MgH_2 during ball milling, producing $NaBH_4$ and the byproduct MgO (Equation (2)), and $NaBH_4$ could then be purified with ethylenediamine and the products could be separated. In order to recycle the byproduct MgO, a method to produce MgH_2 from MgO should be found. This requirement is met by Mg, which is produced industrially by electrolysis of fused magnesium chloride (Equations (5) and (6)) in the third step:

$$MgO + Cl_2 \rightarrow 1/2O_2 + MgCl_2(900\ ^\circ C) \quad (5)$$

$$MgCl_2 \rightarrow Cl_2 + Mg(710\ ^\circ C) \quad (6)$$

Here, chlorine is used to react with MgO at 900 °C to produce $MgCl_2$, and Mg is obtained by electrolysis of fused $MgCl_2$. Mg obtained from Equation (6) may be hydrogenated to produce MgH_2 according to Equation (7) [18]:

$$Mg + H_2 \rightarrow MgH_2 \quad (7)$$

The energy from the exothermic hydrogenation could be stored and utilized for other purposes. MgH_2 produced in this process can react with $NaBO_2$ again to produce $NaBH_4$.

For convenience, all of the aforementioned reactions are assumed to proceed at constant pressure, and the fuel cell reaction is assumed to occur at constant temperature and pressure. We based the calculation on the assumption that the entire cycle is an ideal path, which means that the extent of reactions is 100% and that all products are completely separated.

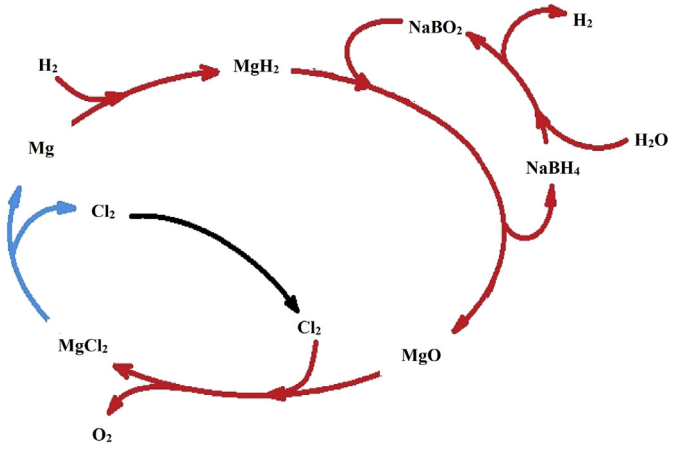

Fig. 2. The path of $NaBH_4$ hydrolysis cycling.

Table 1
Enthalpy change of each reaction in the cycling.

Number (i)	Reaction	Enthalpy change (kJ mol^{-1})
1	$H_2 + 1/2O_2 \rightarrow H_2O$	$\Delta H_1 = -285.83$
2	$1/4NaBH_4 + 1/2H_2O \rightarrow 1/4NaBO_2 + H_2$	$\Delta H_2 = -75.00$
3	$1/4NaBO_2 + 1/2MgH_2 \rightarrow 1/4NaBH_4 + 1/2MgO$	$\Delta H_3 = -65.87$
4	$1/2MgO + 1/2Cl_2 \rightarrow 1/4O_2 + 1/2MgCl_2$ (900 °C)	$\Delta H_4 = -35.26$
5	$1/2MgCl_2 \rightarrow 1/2Cl_2 + 1/2$ Mg (710 °C)	$\Delta H_5 = 298.79$
6	$1/2$ Mg $+ 1/2H_2 \rightarrow 1/2MgH_2$	$\Delta H_6 = -76.15$

According to the standards database [19], the standard enthalpy change of these reactions can be computed as follows:

$$\Delta_r H_m = \sum n \Delta_f H_m \quad (8)$$

where, $\Delta_r H_m$ is the standard enthalpy of reaction, n is the stoichiometric number of each reaction component, and $\Delta_f H_m$ is the standard enthalpy of formation. Therefore, the enthalpy change of the reaction may be computed through Kirchhoff's formula:

$$\Delta H_m = \Delta_r H_m + \int Cp\, dT \quad (9)$$

where, ΔH_m is the enthalpy of reaction, Cp is the heat capacity at constant pressure, and T is the absolute temperature. Because all of the reactions proceed at constant pressure, the thermal effect of the reaction is equivalent to enthalpy change.

In the cycle, the energy released by the exothermic reaction may be collected for recycling. This part of the energy is thus ignored in the calculation and is regarded as heat released from the reaction. However, energy that the endothermic reaction absorbs is the energy input from the environment, which may be computed from the enthalpy change.

The Gibbs free energy is the maximum amount of non-expansion work that can be extracted from the reaction at constant temperature and pressure [20]. Hydrogen reacts with oxygen in fuel cells at constant temperature and pressure, resulting in energy release; thus, the Gibbs free energy of this reaction is the maximum amount of energy that can be extracted. The standard Gibbs free energy of water is the maximum amount of E_o of the cycle. The energy efficiency (η) of $NaBH_4$ hydrolysis and regeneration is thus given by the formula:

$$\eta = E_o/E_i \quad (10)$$

To standardize the unit of measure, the stoichiometric amount of reactants is determined by the number required to generate of 1 mole of hydrogen. For example, 1/4 mole of $NaBH_4$ as reactant can produce 1 mole of H_2, according to Equation (1); thus, the stoichiometric number of $NaBH_4$ is 1/4.

Table 1 shows the enthalpy change of each reaction in the cycle. Equation (7) describes the endothermic reaction of this cycle.

However, purification of $NaBH_4$ entails the evaporation of ethylenediamine, which is endothermic. The enthalpy of vaporization that generates 1 mole of hydrogen is 33.34 kJ, here referred to as ΔH_7.

According to Fig. 2, hydrogen used to produce MgH_2 could not be regenerated in this cycle. Therefore, additional energy input into the cycle is due to hydrogen, and E_i is described by Equation (11):

$$E_i = |1/2\Delta H_1| + \Delta H_5 + \Delta H_7 = 475.05\ \text{kJ} \quad (11)$$

where, ΔH_1 is the enthalpy of Equation (4), and ΔH_5 is the enthalpy of Equation (6).

The Gibbs free energy of the reaction between H_2 and O_2 is −237.1 kJ mol^{-1}, according to Equation (12):

$$H_2 + 1/2O_2 \rightarrow H_2O,\ \Delta G = -237.1\ \text{kJ mol}^{-1} \quad (12)$$

E_o of the cycle is also the Gibbs free energy (ΔG) from Equation (12). E_o is expressed as follows:

$$E_o = |\Delta G| = 237.1\ \text{kJ} \quad (13)$$

According to Equation (10), the maximum energy efficiency is 49.91%, indicating that about half of the E_i in the cycle is the output in each cycle. If the heat released by the exothermic reaction can be collected for recycling, then the maximum energy efficiency is further improved. It was reported that MgO could be reduced to Mg in a solar furnace [21], which is environmentally friendly and is able to improve the maximum energy efficiency. Therefore, according to the maximum energy efficiency that was computed, the cycle is feasible.

3.3. Synthesis of $NaBH_4$ using H–Mg_3La

Hydrogenation using Mg is relatively difficult, but Mg_3La easily undergoes hydrogenation at room temperature. In contrast to the method that uses MgH_2 to produce $NaBH_4$, regeneration by H–Mg_3La may reduce the energy consumption. Therefore, we used H–Mg_3La and $NaBO_2$ to regenerate $NaBH_4$ by ball milling. Fig. 3 shows the XRD pattern of hydrogenated Mg_3La. The peaks could be indexed to MgH_2, LaH_3 and MgO, the last of which was found in small amounts. These results indicate that the Mg_3La phase reacted with hydrogen and transformed into MgH_2 and LaH_3, and that production of MgO was due to the oxidization of Mg_3La. The reaction may be described as follows:

$$LaMg_3 + 9/2H_2 \rightarrow 3MgH_2 + LaH_3 \quad (14)$$

Fig. 4(a) shows the XRD patterns of the reactant mixture before ball milling. Its peaks could be indexed to LaH_3, $NaBO_2$, MgO, and MgH_2. Fig. 4(b)–(f) shows the XRD patterns of the product after 0.5, 3, 5, 10, and 20 h of ball milling, respectively. Peaks of the XRD pattern in Fig. 4(c) are in a good agreement with the characteristic spectrum of LaH_3 crystal, MgO crystal, and MgH_2 crystal in the JCPDS database. The decrease in intensity of the MgH_2 peaks suggests that $NaBO_2$ began to react with MgH_2 and formed $NaBH_4$. The absence of $NaBO_2$ peaks in Fig. 4(c) was due to pulverization of

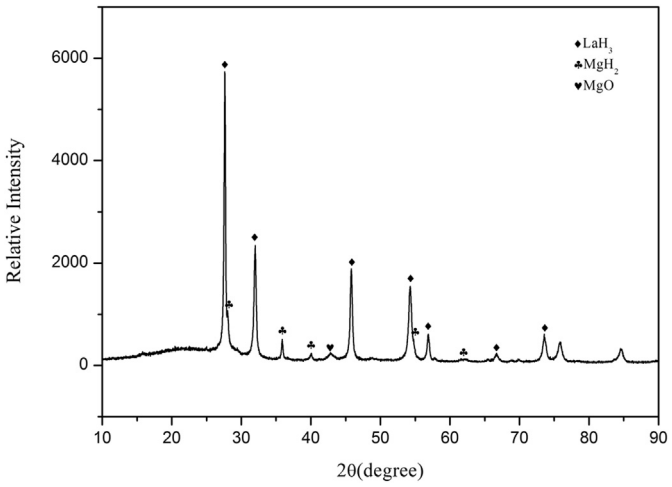

Fig. 3. XRD patterns of hydrogenated Mg_3La.

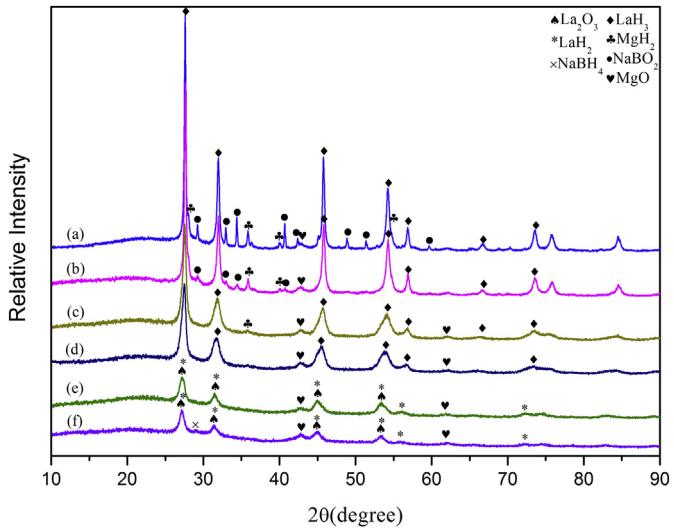

Fig. 4. XRD patterns of (a) the $NaBO_2$–Mg_3La hydride mixture and the product after ball milling the $NaBO_2$–Mg_3La hydride mixture for (b) 0.5 h (c) 3 h (d) 5 h (e) 10 h (f) 20 h.

$NaBO_2$ particles after ball milling and their participation of the particles in the mechanochemical reaction producing $NaBH_4$.

Peaks of the XRD pattern in Fig. 4(d) could be indexed to LaH_3 and MgO. Peaks corresponding to MgH_2 became indistinguishable and those of LaH_3 broadened. Peaks of the XRD pattern in Fig. 4(e) could be indexed to La_2O_3, LaH_2, and MgO. After 10 h of milling, peaks corresponding to MgH_2 were absent and those corresponding to La_2O_3 and LaH_2 appeared in the XRD patterns. These changes indicate that initially, MgH_2 reacted completely with $NaBO_2$. Part of the LaH_3 reacted with the remaining $NaBO_2$ to form $NaBH_4$, and another part transformed to LaH_2. The reaction between $NaBO_2$ and LaH_3 may be expressed as follows:

$$9NaBO_2 + 12MgH_2 + 4LaH_3 \rightarrow 9NaBH_4 + 2La_2O_3 + 12MgO \quad (15)$$

Products of the above reaction were analyzed by FT-IR spectroscopy. Fig. 5 presents the FT-IR spectrum of the product. As shown in Fig. 5, peaks between 2200 to 2400 cm^{-1} and at 1125 cm^{-1} are due to the B–H stretching and B–H deformation vibrations of pure $NaBH_4$, respectively [22]. These peaks confirm

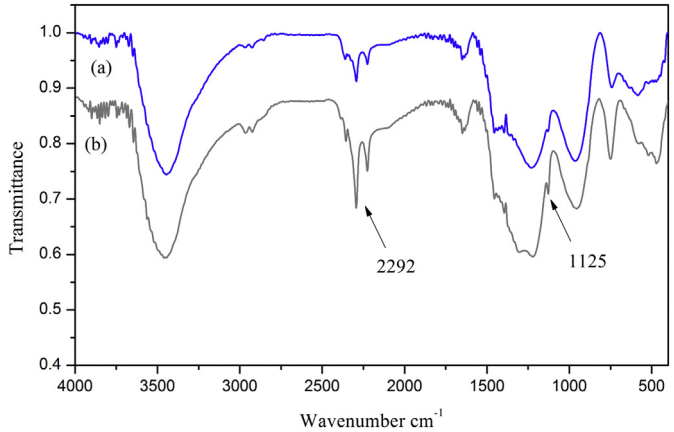

Fig. 5. FT-IR spectrum of the product after ball milling the $NaBO_2$–Mg_3La hydride mixture for (a) 5 h (b) 10 h.

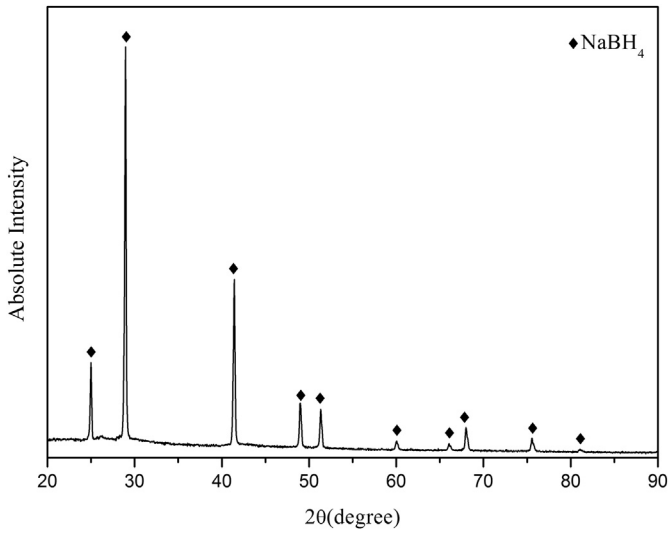

Fig. 6. XRD patterns of the white solid product obtained after purifying.

the reaction of H–Mg_3La with $NaBO_2$ to form $NaBH_4$. Peaks of $NaBH_4$ are relatively weak and even unidentified. Fig. 4(e) also indicates the amorphization of $NaBH_4$ during regeneration. Fig. 4(f) depicts the XRD pattern of the reaction products milled for 20 h. Peaks in this pattern could be indexed to LaH_2, La_2O_3, MgO, and $NaBH_4$. Relatively strong peaks of $NaBH_4$ in Fig. 4(f) are due to crystallization of $NaBH_4$ after 20 h of milling. XRD patterns of the white solid product obtained after 20 h of milling and purification are shown in Fig. 6. Peaks in this pattern could be indexed to the $NaBH_4$ crystal. Thus, these results also prove that H–Mg_3La could react with $NaBO_2$ to produce $NaBH_4$.

4. Conclusion

Recycling of $NaBH_4$ by $NaBH_4$ hydrolysis and $NaBO_2$ regeneration using a reaction with MgH_2, was examined in the present study. The energy efficiency of the entire recycling process was determined according to thermodynamics calculations. The maximum energy efficiency (49.91%) indicates that reaction of MgH_2 with $NaBO_2$ to form $NaBH_4$ during ball milling is feasible. An inexpensive method of $NaBH_4$ regeneration by ball milling was demonstrated by reacting H–Mg_3La with $NaBO_2$. Mg_3La, which could be hydrogenated at room temperature, further improved the energy efficiency. In the reaction of H–Mg_3La with $NaBO_2$, MgH_2 reacts with $NaBO_2$ completely in the first step and then LaH_3 reacts with $NaBO_2$ to form $NaBH_4$.

Acknowledgments

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